# Characterization of Light Absorption and Light Scattering Properties of Particulate Samples Using Temporally and Spatially Resolved Diffuse Reflectance Measurements

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# Our Work

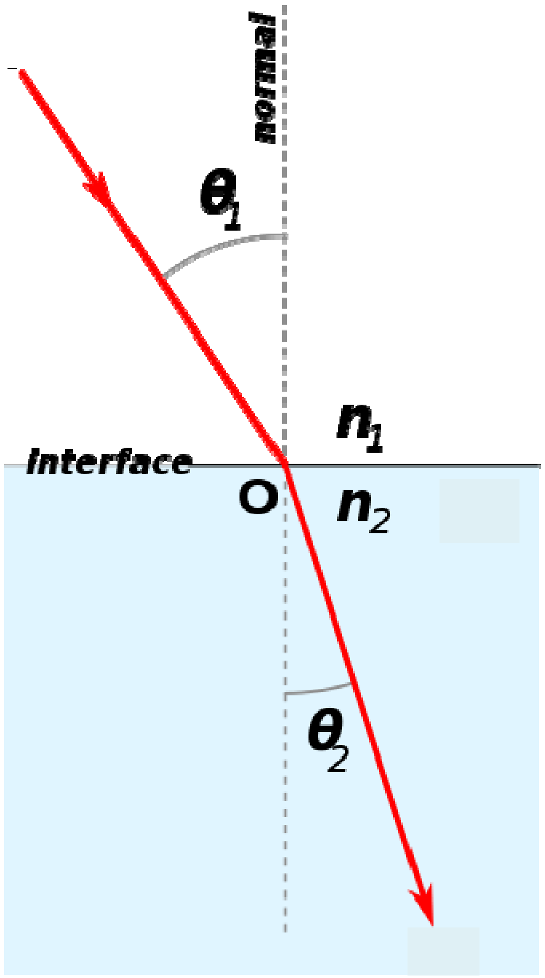
- Kinetic modeling of chemical batch reactions of slurries
  - Light absorption and light scattering are confounded
  - Separating these effects and using them to gain quantitative information is a challenge

# Light Absorption and Scattering

- Light can be absorbed, reflected, refracted, scattered, or transmitted through a sample
- Absorption is wavelength dependent
- Scattering is ALSO wavelength dependent
- All of these effects are dependent on the angle of incidence and the indices of refraction

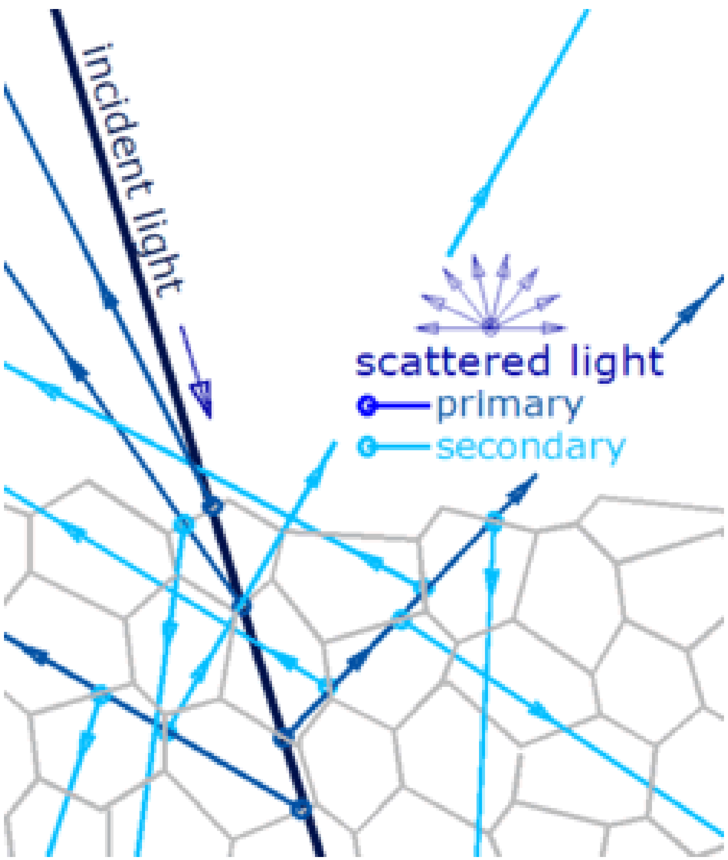
$$n_1 sin\theta_1 = n_2 sin\theta_2$$

(ex. refraction)




Source: Internet

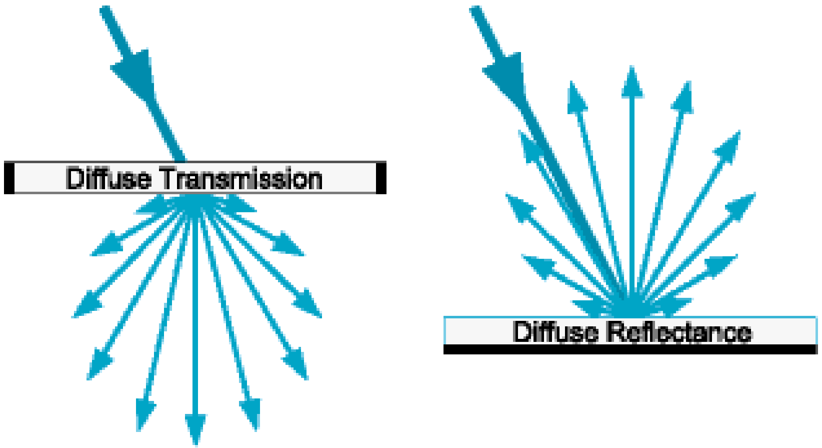
# Challenges




Source: Internet

- Making light absorption measurements of a slurry is difficult
  - Hard to discern differences in absorption and scattering
  - Sample is constantly changing

# What Is Diffuse Reflectance?




Diffuse transmission and reflectance.

- Light that reflects back from a sample is measured
- Less labor intensive than diffuse transmission
- Absorbance is calculated based on the light that returns to the detector

Picture Source: Internet

# Determination of Optical Properties

- Diffuse Reflectance can be used to calculate the absorption coefficient ($\mu_a$) and the reduced scattering coefficient ($\mu_s$')
  - $\mu_s$' incorporates the scattering coefficient ($\mu_s$) with the anisotropy (g) of the sample
    - Anisotropy refers to the directional differences of the sample

$$\mu_s' = \mu_s(1 - g)$$

# Four Frequently Used Methods

- Temporally Resolved
- Spatially Resolved
- Integrating Sphere
- Frequency Domain
  - Determines the optical properties from measured amplitude attenuation and phase shift of the propagating photon density wave

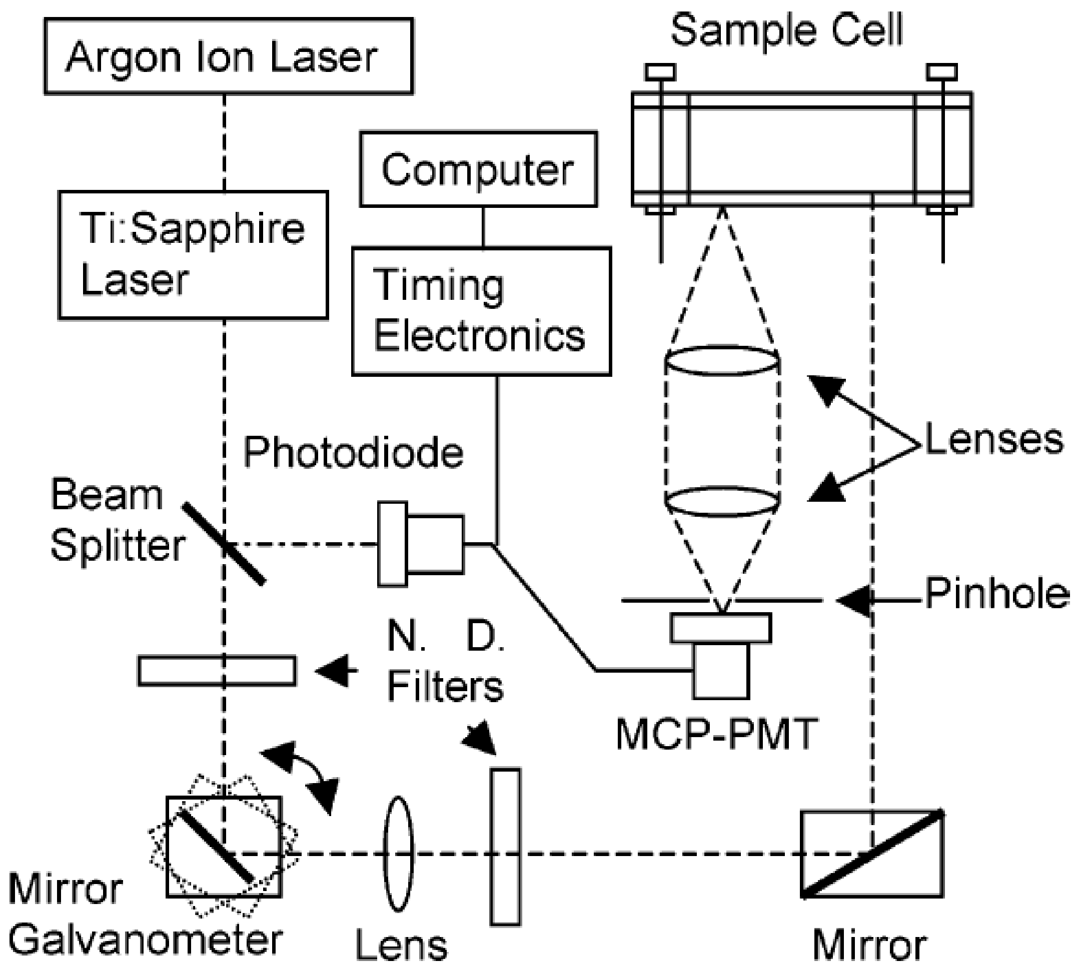
# Power Law Analysis Estimates of Analyte Concentration and Particle Size in Highly Scattering Granular Samples from Photon Time-of-Flight Measurements

Pandozzi F. and Burns D.
McGill University
2007

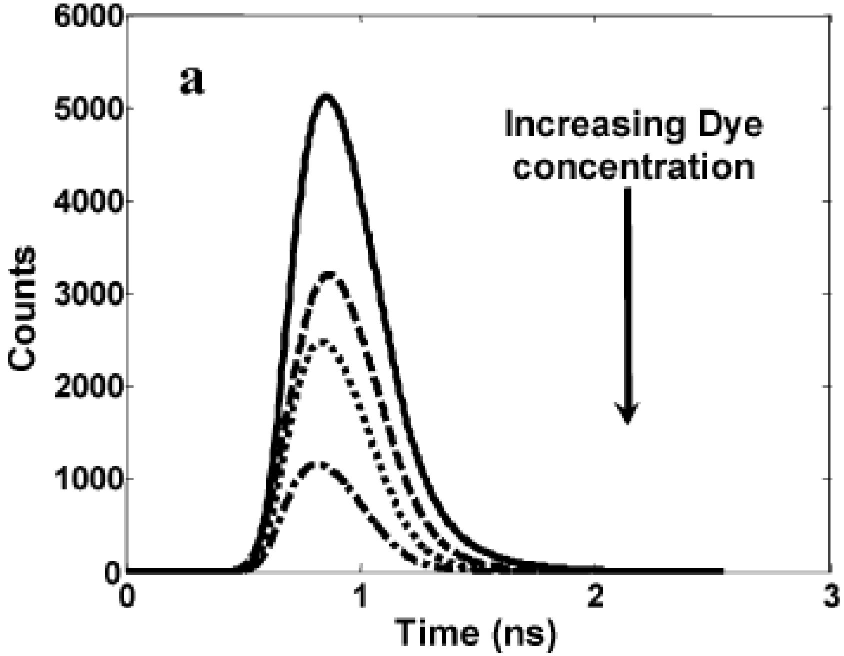
# Introduction

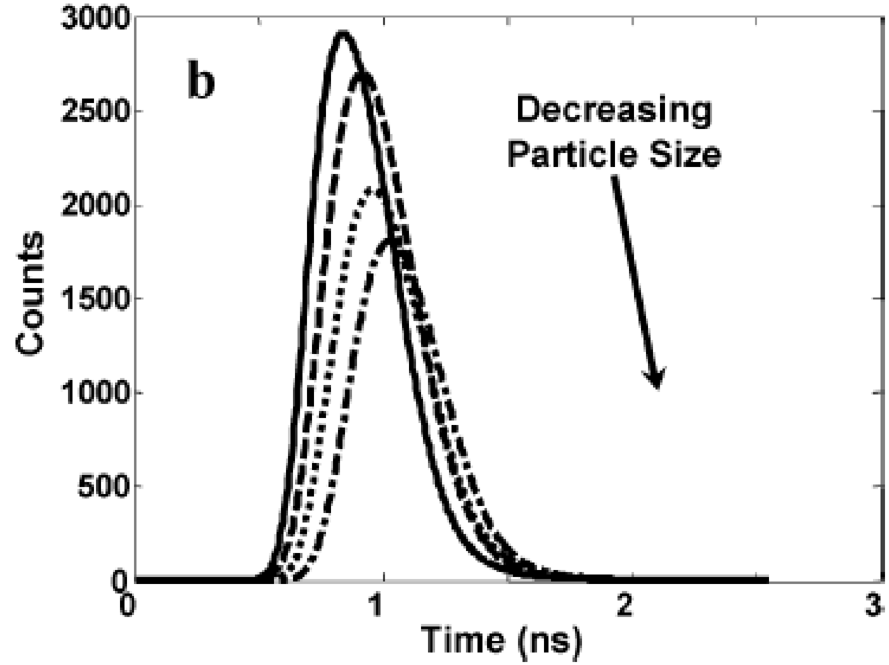
- Photon Time-of-Flight spectroscopy (PTOFS)
- Power Law analysis
- Calculated $\mu_a$ and the particle size from silica gel samples
  - Samples varied in average particle diameter and dye amounts
  - Individual properties were held constant during measurement

# Photon Time-of-Flight Spectroscopy



# Photon Time-of-Flight Spectroscopy



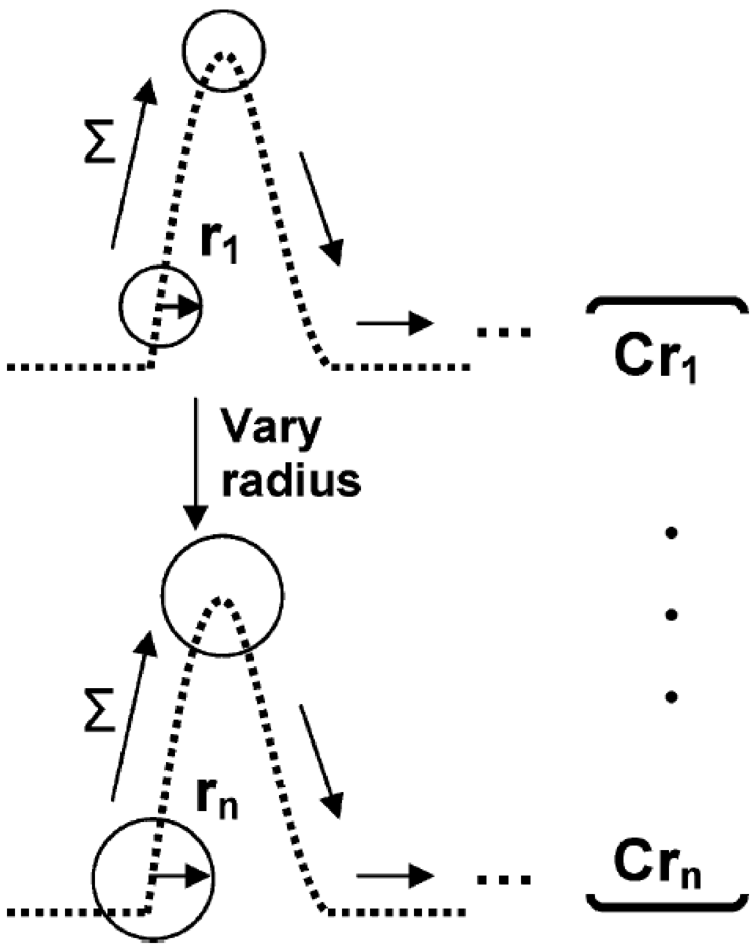



# Power Law Analysis

- A data pre-processing technique coupled with a data mining technique
- Examines self-similarities of data at different scales

$$C_r = \frac{\sum_{i=1}^{N} (\text{total number of points within } r \text{ of point } x_i)}{N(N-1)}$$

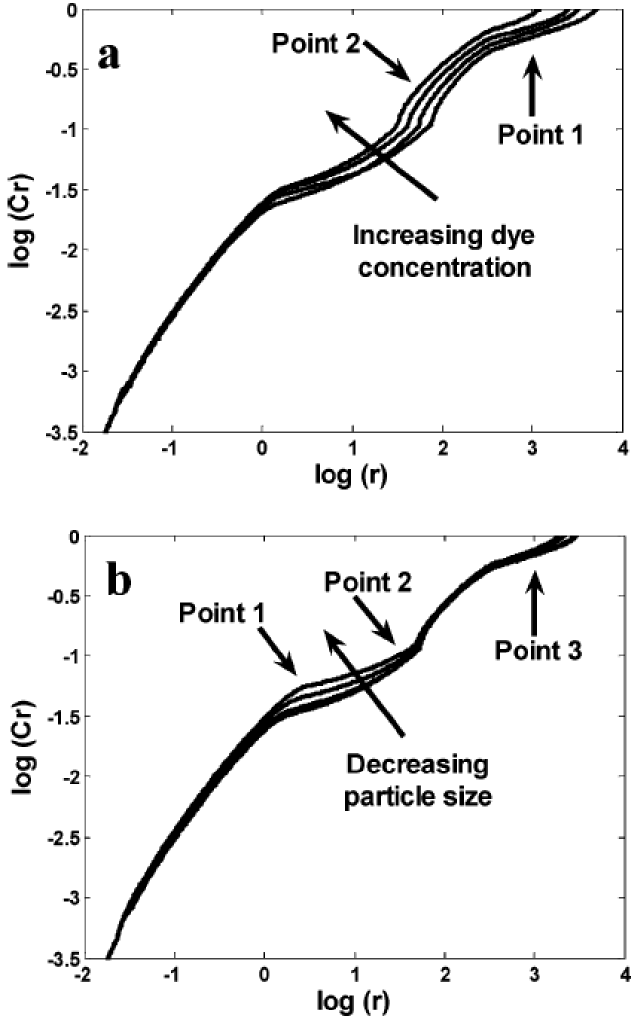
N = total number of data points



# Results

- Correlation Dimension Plots
- Stepwise Multiple Linear Regression (SMLR) analysis was used to determine the best areas of correlation
- Multiple Linear Regression (MLR) models were built from the slope and intercept information from the highly correlated regions

$$Y = b_0 + b_1x_1 + \cdots + b_mx_m$$



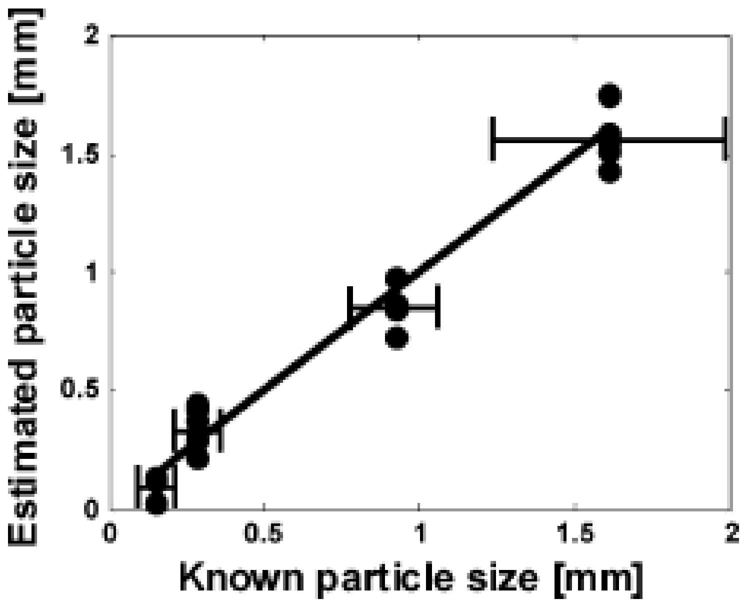
# Results

**Table 1. Results from MLR Analysis for the Estimation of Absorption Coefficients**

| separation (mm) | parameters used | $R^2$ | CV (%) |
|---|---|---|---|
| 5 | $m$, $b_y$, $b_x$, $A$ | 0.93 | 25.5 |
| 15 | $m$, $b_y$, $A$ | 0.92 | 26.5 |
| 15 | $m$, $b_y$ | 0.92 | 27.3 |
| 15 | $b_x$ | 0.88 | 33.0 |

**Table 2. Regression Results for the Estimation of Particle Size Using MLR**

| separation (mm) | parameters used | $R^2$ | CV (%) |
|---|---|---|---|
| 5 | $m$, $b_y$, $b_x$, $A$ | 0.97 | 13.4 |
| 5 | $m$, $b_y$, $A$ | 0.97 | 13.1 |
| 5 | $m$, $b_y$ | 0.95 | 16.4 |
| 5 | $m$ | 0.95 | 16.8 |




$m$= slope
$b_x$= x-intercept
$b_y$= y-intercept
A= PTOFS peak area

# Fiber-Optic Probe for Noninvasive Real-Time Determination of Tissue Optical Properties at Multiple Wavelengths

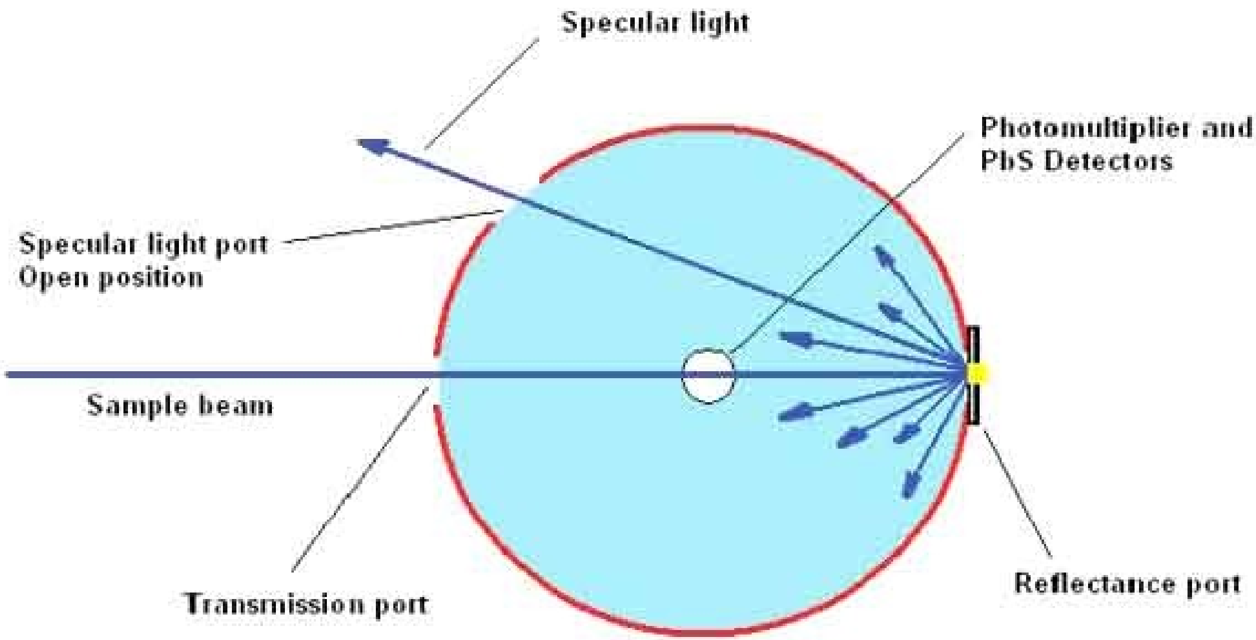
Dam J., Pedersen C., et al.

2001

# Introduction

- Use spatially resolved diffuse reflectance to accurately calculate the $\mu_a$ and $\mu_s'$ values for intralipid-dye phantoms
  - Intralipid concentrations: 0.6, 0.8, … ,1.6%
  - Ink concentrations: 0.0, 0.2, … , 1.2%
- Compare results to those from an integrating sphere method

# Integrating Sphere



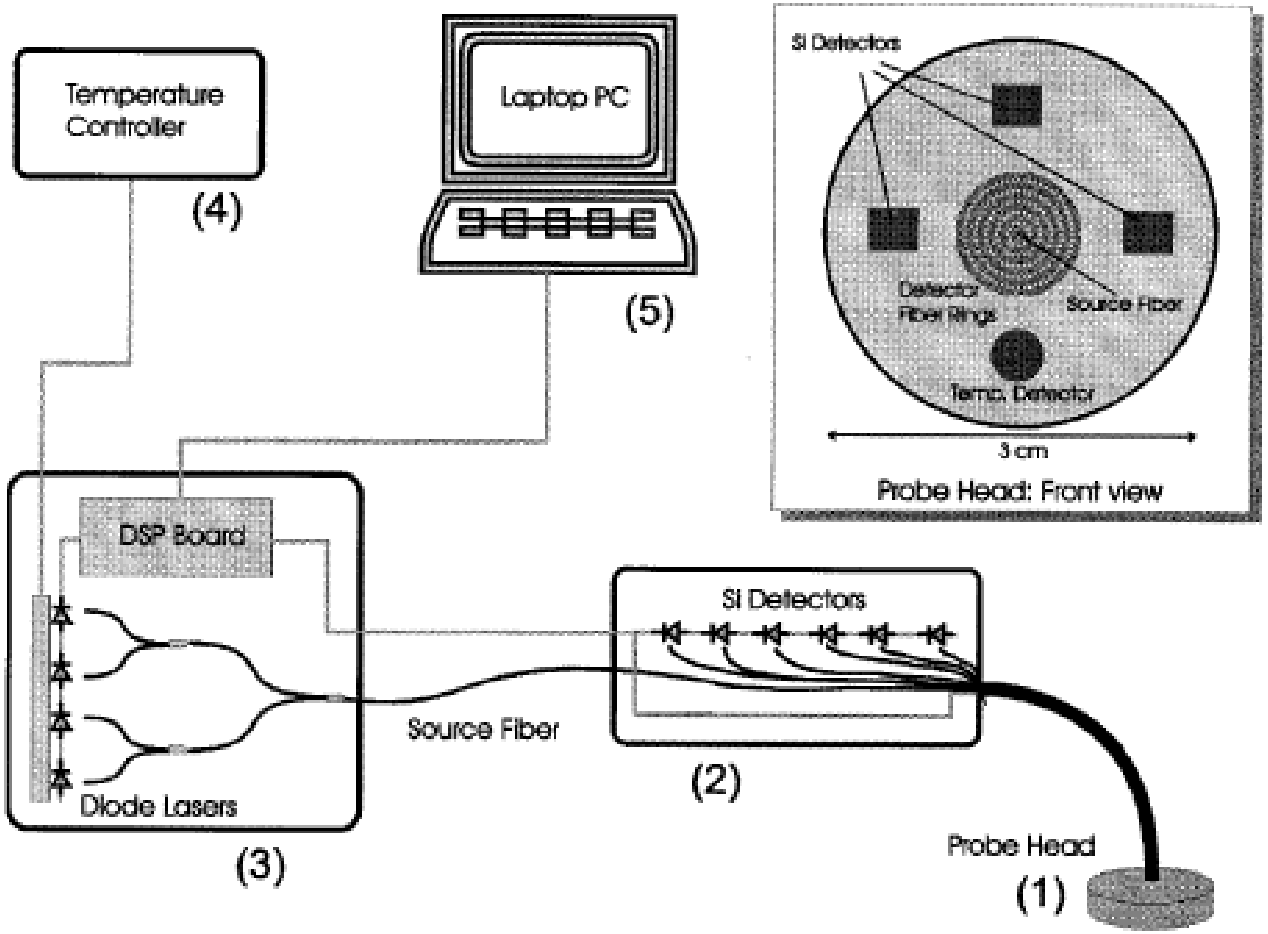

Integrating Sphere
Diffuse Reflectance Mode

Wei-Boon, T. "Measuring Solar Energy Properties of Architectural Glass, Mirrors and Photovoltaic Materials and Thermal Emissivity of Materials." PerkinElmer.

# Spatially Resolved Measurements

- Uses a light source of constant intensity with multiple detectors at different distances from the source
- Less complicated in design, less time consuming, and equipment is cheaper
- 2 primary forms:
  - Contact detection
  - Noncontact detection

# Spatially Resolved Probe



# Multiple Polynomial Regression

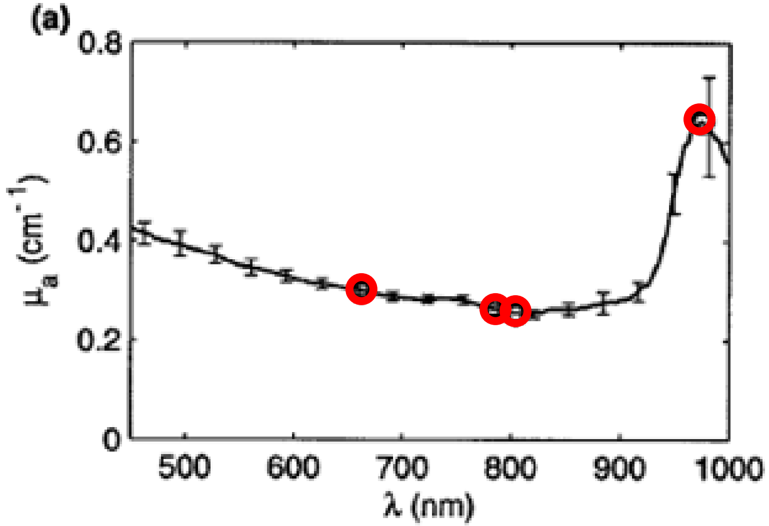
- $\mu_a$ and $\mu'_s$ were calculated using a multiple polynomial regression method

$$R_{1,\text{fit}}(\mu_a, \mu_s', m) = a_0 + a_1\mu_a + a_2\mu_a^2 + \cdots a_m\mu_a^m) \times (b_0 + b_1\mu_s'^2 + \cdots b_m\mu_s'^m)$$

$$R_{2,\text{fit}}(\mu_a, \mu_s', m) = c_0 + c_1\mu_a + c_2\mu_a^2 + \cdots c_m\mu_a^m) \times (d_0 + d_1\mu_s'^2 + \cdots d_m\mu_s'^m)$$

- Then the Newton-Raphson algorithm was used to calculate the coefficients

# Results



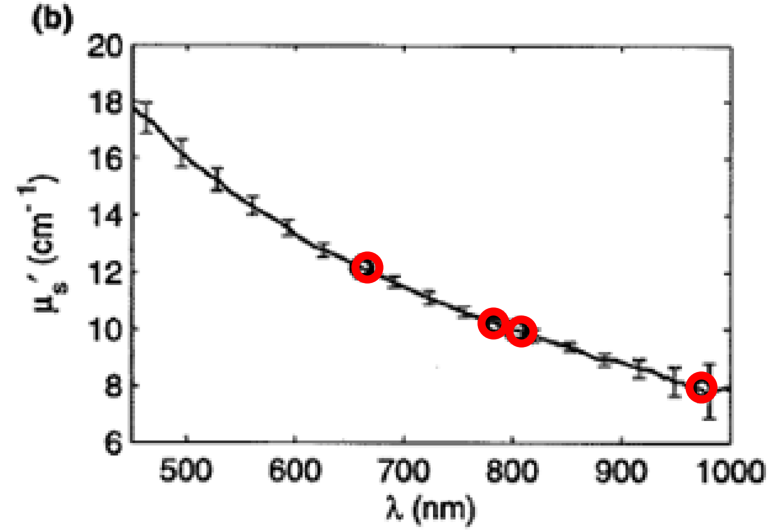




**Table 2. Leave-One-Out Cross-Validation Prediction Tests Based on Phantom Measurements[a]**

| | Prediction Errors (%) | | | |
|---|---|---|---|---|
| | $\mu_a$ | | $\mu_s'$ | |
| λ (nm) | Mean | Max | Mean | Max |
| 600 | 3.3 (2.5) | 18 (6.7) | 1.7 (1.7) | 3.7 (4.0) |
| 785 | 2.8 | 8.3 | 1.3 | 3.4 |
| 805 | 2.6 | 9.0 | 1.5 | 4.2 |
| 974 | 3.7 | 9.7 | 1.6 | 3.8 |

# Comparison of Spatially and Temporally Resolved Diffuse-Reflectance Measurement Systems for Determination of Biomedical Optical Properties

Swartling D., Dam J., and Andersson-Engels S.
Lund Institute of Technology
2003

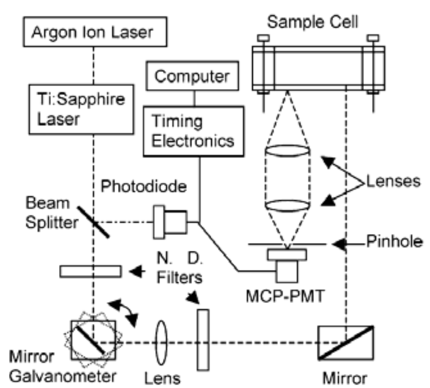
# Goals

- Compare the results of both temporally and spatially resolved methods and investigate their limitations
- The two methods were compared to results from an integrating sphere method
- Used epoxy resin tissue phantoms, in vitro, and in vivo samples for testing

# Probes

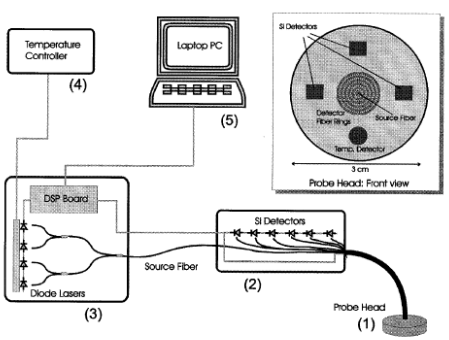
**TEMPORALLY RESOLVED**

- Similar to the PTOFS system
- A form of the radiative transport equation was used in order to calculate coefficient values



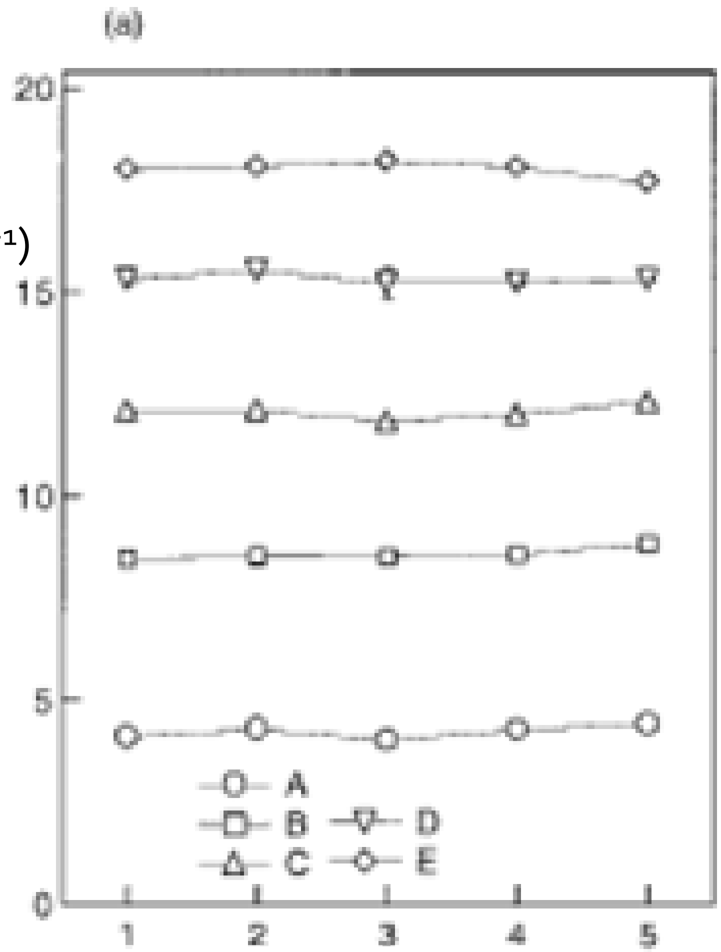

**SPATIALLY RESOLVED**

- Used the same probe as mentioned previously
- Multiple Polynomial Regression was used to relate the results to the coefficient values



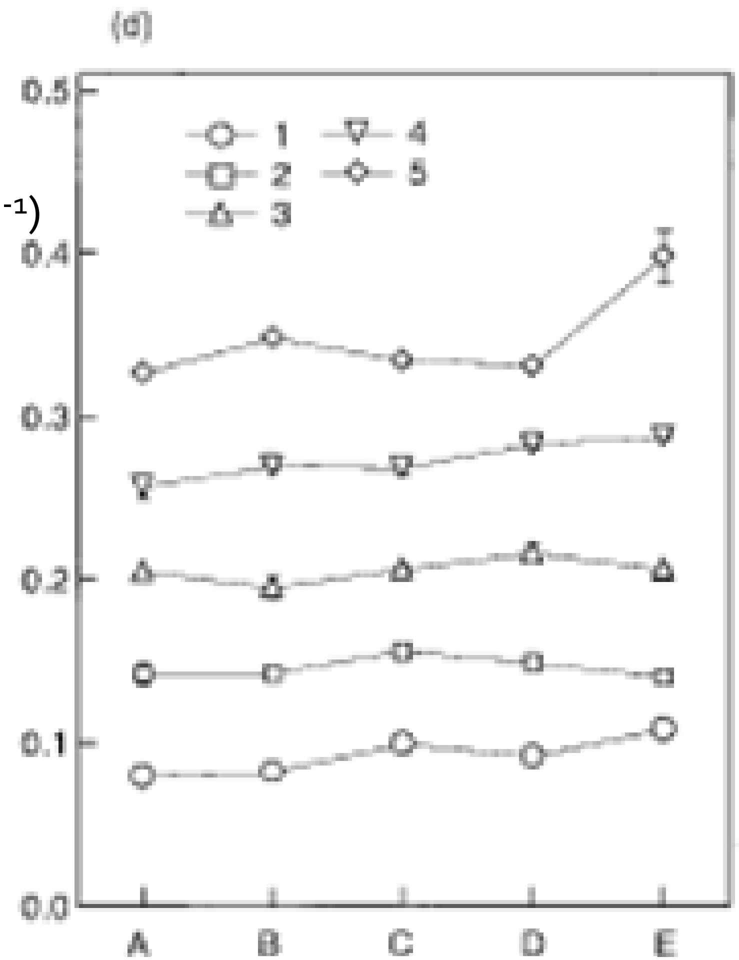
# Spatially Resolved Results of Tissue Phantoms

$\mu'_s$ ($cm^{-1}$)




Increasing $\mu_a$: Increasing dye conc.

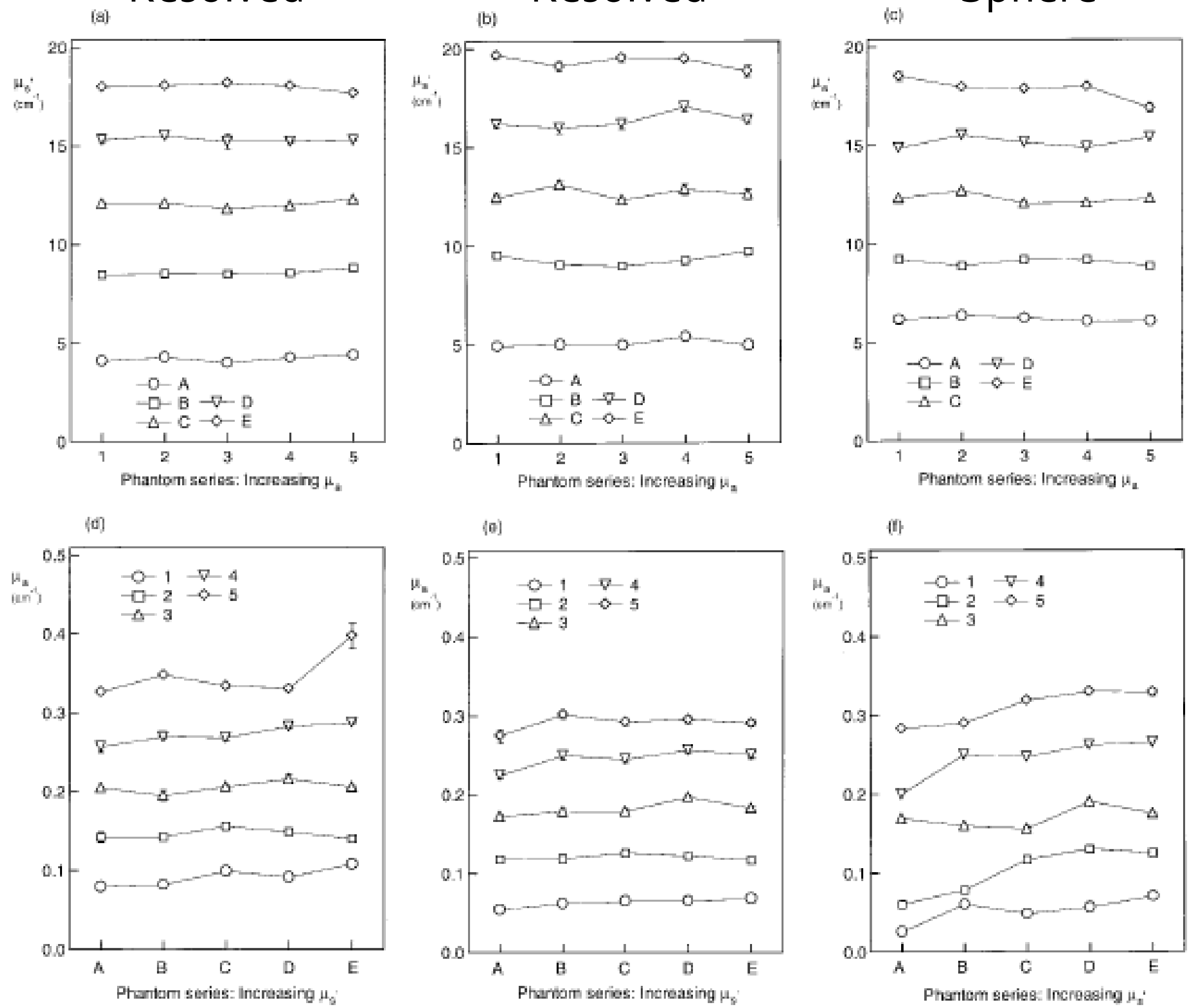
$\mu_a$ ($cm^{-1}$)




Increasing $\mu'_s$: Increasing TiO conc.

# Phantom Results for All Methods

Spatially Resolved | Temporally Resolved | Integrating Sphere



# In Vitro and In Vivo Results

**Table 2. Results of Measurements of the Meat Sample**

| Property | Fiber-Probe Method $\mu_s'$ ($cm^{-1}$) | Fiber-Probe Method $\mu_a$ ($cm^{-1}$) | Time-Resolved Method $\mu_s'$ ($cm^{-1}$) | Time-Resolved Method $\mu_a$ ($cm^{-1}$) | Integrating-Sphere Method $\mu_s'$ ($cm^{-1}$) | Integrating-Sphere Method $\mu_a$ ($cm^{-1}$) | Integrating-Sphere Method $g$ | Integrating-Sphere Method $\mu_s$ ($cm^{-1}$) |
|---|---|---|---|---|---|---|---|---|
| Mean | 6.9 | 0.043 | 6.6 | 0.037 | 4.9 | 0.061 | 0.94 | 82 |
| Standard deviation | 1.4 | 0.014 | 0.5 | 0.008 | 1.1 | 0.020 | 0.006 | 11 |

**Table 3. Results of Measurements of Forearms of the Experimenters**[a]

| Person | Fiber-Probe Method $\mu_s'$ | Time-Resolved Method $\mu_s'$ ($cm^{-1}$) |
|---|---|---|
| 1 | 10.6 | 2.5 |
| 2 | 11.8 | 5.0 |

[a]For both persons, the fiber-probe method measured $\mu_a$ as 0.19 cm, and the time-resolved method measured $\mu_a$ as 0.20 cm.

# Final Conclusions

- Advantages:
  - Studies have shown great potential for these measurement methods in numerous applications

- Disadvantages:
  - Methods are limited by the samples being measured and the mathematical models used to describe the measurements

# References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# Diffusion Theory Model

- Used a steady state diffusion theory model to describe the spatial diffuse reflectance and for optical property estimation
  - Model is derived from the radiative transport equation (Boltzmann equation) which describes the flow of photons through a scattering system
- From the equation $\mu_a$ and $\mu_s'$ were calculated for all wavelengths
  - Equation was used to compensate for the change in reflectance due to the curvature of the sample fruit or vegetable